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U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER
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DESIGNATED/ELECTED OFFICE (DO/EO/US)
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U.S. APPLICATION NO. (If known, see 37 CFR 4.5)

09/601946

INTERNATIONAL APPLICATION NO.
PCT/EP98/05150INTERNATIONAL FILING DATE
13 August 1998 13.08.98PRIORITY DATE CLAIMED
09 February 1998 (09.02.98)

TITLE OF INVENTION PROCESS FOR PREPARING LITHIUM TRANSITION METALLATES

APPLICANT(S) FOR DO/EO/US M. Benz; W. Kummer; E. Pross; J. Schmoll; W. Schweda;
D. Duff; R. Leiberich; C. Schild

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(3)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☐ A FIRST preliminary amendment.
☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☒ A change of power of attorney and/or address letter.
16. ☒ Other items or information: WO 99/40029 publication, search report in German, copy of original PCT Request (in German), IB 306 copies of prior art references

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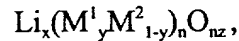
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Process for preparing lithium transition metallates

The present invention relates to a process for preparing lithium transition metallates of the general formula

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wherein

10 M¹ represents nickel, cobalt or manganese,

M² represents a transition metal which is different from M¹ and is chromium, cobalt, iron, manganese, molybdenum and/or aluminium,

15 n is 2 if M¹ is manganese, and n is 1 if M¹ is nickel or cobalt, wherein

x has a value from 0.9 to 1.2,

y has a value between 0.5 and 1 and

20

z has a value between 1.9 and 2.1.

These types of lithium transition metallates are used as electrode materials, in particular as cathode materials for non-aqueous lithium storage battery systems, so-called lithium ion batteries.

A number of proposals have already been made relating to methods of preparation of these types of lithium transition metallates, but these are mostly unsuitable for large-scale production or lead to products which have imperfect electrochemical properties.

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The use of LiCoO_2 has recently gained acceptance, but this is extremely expensive due to the limited availability, and thus high price, of cobalt and is therefore not suitable for mass production (e.g. to provide the power for electrically operated vehicles). Therefore intensive efforts have already been made to replace all or some of the LiCoO_2 with, for example, LiNiO_2 and/or LiMn_2O_4 as a cathode material.

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Synthesis of the corresponding cobalt compound LiCoO_2 is generally regarded as a non-critical procedure. Due to the thermal stability of LiCoO_2 , it is even possible, with this system, to react cobalt carbonate and lithium carbonate, as reaction components, directly at relatively high temperatures without troublesome concentrations of carbonate being left in the final product.

The transfer of this method to LiNiO_2 has been possible only at temperatures of 800°C to 900°C . These high calcination temperatures, however, lead to partly decomposed lithium nickelates with relatively low storage capacities and/or unsatisfactory resistance to cyclic operation.

For this reason, carbonate-free mixtures are proposed for preparing LiNiO_2 , in which, in most cases, β -nickel hydroxide is favoured as the nickel component, such as is described, for example in US-A 5 591 548, EP 0 701 293, J. Power Sources 54 (95) 209-213, 54 (95) 329-333 and 54 (95) 522-524. Moreover, the use of nickel oxide was also recommended in JP-A 7 105 950 and that of oxynickel hydroxide NiOOH in DE-A 196 16 861.

According to US-A 4 567 031, the intimate mixture is prepared by co-precipitation of soluble lithium and transition metal salts from solution, drying the solution and calcining. Relatively finely divided crystals of the lithium transition metallate are obtained in this way at comparatively low calcining temperatures and within comparatively short times. The allocation of lithium and transition metal ions to particular layers in the crystal lattice, however, is greatly distorted so that, to a large extent, nickel ions occupy lithium layer lattice positions and vice versa. These types of crystals have unsatisfactory properties with regard to their use as electrodes in rechargeable batteries. Other processes (EP-A 205 856, EP-A 243 926, EP-A 345 707) start with solid, finely divided carbonates, oxides, peroxides or hydroxides of the initial metals. The intimate mixture is prepared by joint milling of the starting metals. The formation of lithium transition metallates takes place by solid diffusion during calcination. Solid diffusion requires comparatively high temperatures and comparatively long calcining times and does not generally lead to phase-pure lithium metallates with outstanding electronic properties. Extensive observations appear to prove that, in the case of the nickel system, decomposition of LiNiO_2 with the

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production of Li_2O and NiO is initiated during prolonged thermal treatment at temperatures above about 700°C .

Therefore, in order to intensify the intimate mixing procedure, it has already been proposed, according to EP-A 468 942, to start the preparation of lithium nickelate with powdered nickel oxide or hydroxide, suspending the powder in a saturated lithium hydroxide solution and extracting the water from the suspension by spray drying. This should lead to a reduction in the calcining time and calcining temperature. Due to the relatively low solubility of lithium hydroxide in water, however, the homogeneity of this mixture is limited.

US-A 5 591 548 proposes milling a powdered oxygen-containing transition metal compound with lithium nitrate and then calcining under an inert gas. The advantage of this process is the low melting point of lithium nitrate, 264°C , which means that intimate mixing takes place after heating to, for example, 300°C in the form of a suspension of transition metal particles in molten lithium nitrate, which favours reaction with the solid.

The disadvantage of this process is that, during calcination, the gases released (H_2O , NO_x , O_2) do not escape, or escape only very slowly, from the viscous molten suspension so that the intimate contact required for the solid reaction and diffusion is hindered and on the other hand only a few suspended particles are present due to concentration inhomogeneities in the geometric spacing. Therefore, interruptions in the calcining process and intermediate milling to homogenise the reaction material are required.

Accordingly, it would be desirable to perform calcination in a moving bed, which would have a beneficial effect on release of the gases produced during reaction, product homogeneity and the residence time required. However, the use of a moving bed conflicts with the use of low-melting lithium compounds such as lithium nitrate or lithium hydroxide because these would then form the expected viscous molten suspension with the transition metal compound and caking would occur at the limiting walls of the moving bed and the product would become agglomerated due to the production of this suspension during the course of reaction.

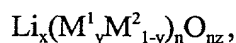
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It has now been found that agglomeration of the product and caking at the limiting walls of the moving bed can be avoided if the transition metal compound is used in the form of a powder with a specific surface area of at least 10 m²/g (BET), wherein, before calcination, the transition metal compound with a large specific surface area is
 5 impregnated with the solution of an oxygen-containing lithium compound and the solvent is removed by drying.

As a result of the high specific surface area, the transition metal compound powder is able to absorb the lithium compound in such a way that a continuous phase cannot be
 10 produced on heating to a temperature above the melting point of the lithium compound and caking of the transition metal compound powder which is coated with the lithium compound, with the wall of the reactor as well as of the powder particles with each other, is very largely suppressed.

15 Accordingly, the invention provides a process for preparing lithium transition metallates of the general formula



20 wherein

M¹ represents nickel, cobalt or manganese,

M² represents chromium, cobalt, iron, manganese, molybdenum or aluminium and
 25 is not identical to M¹,

n is 2 if M¹ is manganese, otherwise 1,

x is a number between 0.9 and 1.2,
 30

y is a number between 0.5 and 1.0 and

z is a number between 1.9 and 2.1,

35 by calcining an intimate mixture of oxygen-containing transition metal compounds and an oxygen-containing lithium compound, which has been obtained by treating a solid

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powdered transition metal compound with a solution of the lithium compound and drying, characterised in that at least the M^1 compound is used in the form of a powder with a specific surface area of at least $10 \text{ m}^2/\text{g}$ (BET) and calcination is performed in a moving bed.

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The M^1 compound preferably has a specific surface area of at least $25 \text{ m}^2/\text{g}$, particularly preferably at least $40 \text{ m}^2/\text{g}$.

Hydroxides are used as preferred M^1 transition metal compounds. Nickel hydroxide is particularly preferred. β -nickel hydroxide with a specific surface area of 60 to $80 \text{ m}^2/\text{g}$ is particularly preferably used, especially if it has been obtained as described in US-A 5 391 265.

If y is less than 1, at least some of the M^2 transition metal compound is preferably used in the form of a mixed hydroxide of the formula $(M^1_y M^2_{1-y})(\text{OH})_2$. The value of y should preferably be greater than 0.8, particularly preferably greater than 0.9.

Lithium hydroxide and/or lithium nitrate may be used as oxygen-containing lithium compounds. These are preferably mixed with the transition metal compound in aqueous solution and then dried and granulated. Lithium nitrate is used as the preferred oxygen-containing lithium compound. The aqueous solution of the lithium compound is preferably used in a concentrated form, in the case of lithium nitrate as a more than 35% strength aqueous solution.

According to one variant of the process according to the invention, at least some of the M^2 transition metal compound may be used as a solution constituent in the solution of the lithium compound for impregnating the M^1 transition metal compound.

To prepare the intimate mixture, the solid, powdered transition metal compound is mixed with the solution of the lithium compound, with stirring, and then the solvent, in particular water, is removed by drying, e.g. by spray-drying, fluidised bed spray granulation or mixer agglomeration. A spray dried material with an agglomerate size of less than $100 \mu\text{m}$ is preferred.

Subsequent calcination in a moving bed may be performed in a rotary kiln, a fluidised bed or a fall-shaft reactor (downer). The use of a rotary kiln is particularly preferred.

In this case, the granules are introduced continuously or batchwise into a preferably electrically heated rotary kiln and treated over a residence time of 0.5 to 10 hours, preferably 1 to 5 hours, at a temperature of 500°C to 800°C, preferably 550°C to 650°C, particularly preferably 580°C to 620°C.

When heating the intimate mixture to the calcination temperature, the temperature range from below the melting point of the lithium compound up to the calcination temperature should be traversed as rapidly as possible. Accordingly, the intimate mixture should be introduced into a rotary kiln which has already been preheated to the calcination temperature or into a moving bed which has already been preheated to the calcination temperature.

If lithium nitrate is used as the oxygen-containing lithium compound, the intimate mixture can be preheated to a temperature of up to 200°C, preferably 150°C to 180°C. If lithium hydroxide is used, preheating may take place up to a temperature of 350°C.

Calcination may be performed in an atmosphere which contains up to 50% oxygen, for example air. Calcination is preferably performed, for at least two thirds of the calcination time, under a substantially oxygen-free inert gas, for example argon, with an oxygen content of less than 5%, in particular less than 3%. In this case, the mixture is calcined under an oxygen-containing gas for the remainder of the calcination time. If the moving bed is operated in a batch process, the atmosphere can be exchanged for an oxygen-containing atmosphere after passage of at least two thirds of the calcination time. If a continuously operated rotary kiln is used, an oxygen-containing atmosphere or oxygen may be introduced, preferably in the last third of the kiln, using a lance.

According to the invention, it is also possible to perform post-calcination under an oxygen-containing atmosphere in a separate moving bed.

In the interests of ensuring a narrow distribution of residence times during calcination, batch operation per se is preferred. However, it is also possible to achieve a sufficiently narrow range of residence times with a half-width of less than one quarter of the average residence time in a continuously operated rotary kiln by inserting appropriate baffles with a tapering cross-section in the rotating tube.

Following calcination, the powdered lithium transition metallate emerging from the moving bed is cooled to room temperature (less than 100°C) and subjected to gentle milling. Suitable milling devices are, for example, those which use the shear effect of a high speed gas profile, when crushing is achieved by particle-particle impact, such as fluidised bed counterstream milling or microfluidised milling. Milling is preferably performed (after removal of the fine fraction) down to an average particle size of 15 to 25 µm diameter. According to a particularly preferred embodiment of the invention, the fine fraction from milling is either recycled to the moving bed or mixed with the powdered, oxygen-containing transition metal compound and then treated together with the solution of oxygen-containing lithium compound and dried, i.e. impregnated.

Lithium nitrate is particularly preferably used as the oxygen-containing lithium compound. The NO_x gas released during calcination in this case is preferably absorbed in an aqueous lithium hydroxide solution and the lithium nitrate solution produced is used to impregnate the powdered transition metal compounds.

Fig. 1 is a schematic diagram of a preferred embodiment of the present invention for producing lithium nickelate. The pre-mix production unit A consists of a stirred container, in which a 40% strength aqueous lithium nitrate solution is initially placed, into which is stirred the powdered β-nickel hydroxide with an average particle size of 10 µm and a specific surface area of 65 m²/g. The slurry obtained is dried by spray drying and introduced into rotary kiln B as granules with an average particle diameter of about 100 µm. The contents of the kiln are held at sinter temperature under an inert gas for preferably 1 to 3 hours. Then (with batch operation), the argon atmosphere can be replaced by an atmosphere containing 20 to 50% oxygen. Then the rotary kiln is cooled and the lithium nickelate obtained is milled in a fluidised bed counterstream mill C to a particle diameter of less than 40 µm and the fine fraction with particle sizes of less than 3 µm are separated by air classification or in a cyclone and collected for recycling to kiln B. The NO_x-containing kiln atmosphere is scrubbed with aqueous lithium hydroxide solution in scrubber D and the lithium nitrate obtained is recovered for the production of another premix.

Examples**Example 1**

- 5 A highly porous nickel hydroxide with a specific surface area of about 65 m²/g BET is stirred into an approximately 40% strength aqueous solution of lithium nitrate. The molar ratio of LiNO₃ to Ni(OH)₂ is 1.03. The suspension is dried in a spray drying tower. The dried powder with an average particle size of about 60 µm is mixed with 5 wt.% of lithium nickelate with a particle size of <5 µm.
- 10 500 g of the powder mixture are placed in the hot zone of a laboratory rotary kiln heated to 620°C, through which flows a stream of nitrogen at a speed of 84 m/h. The rotary kiln has an internal diameter of 55 mm and is rotated at 1/4 rpm.
- 15 After one hour, the rotary kiln is cooled to less than 100°C and samples are taken from the kiln.

X-ray diffraction analysis gives the following peak ratios:

20	I_{104}/I_{003} (LiNiO ₂)	0.76
	$I_{111}(\text{Li}_2\text{O})/I_{101}(\text{LiNiO}_2)$	0.038
	Half-width 003 reflection	0.17
	Half-width 104 reflection	0.19

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Example 2

Example 1 is repeated with the difference that the rotary kiln is held at 600°C and cooling takes place after two hours.

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Samples taken after cooling gave the following values:

I_{104}/I_{003} (LiNiO ₂)	1.1
$I_{111}(\text{Li}_2\text{O})/I_{101}(\text{LiNiO}_2)$	0.1

Half-width 003 reflection 0.27

Half-width 104 reflection 0.25

- 5 The majority of the product is post-calcined under air for 16 hours at 620°C in the rotary kiln. The following values were then obtained from X-ray diffraction analysis:

I_{104}/I_{003} (LiNiO₂) 0.59

$I_{111}(\text{Li}_2\text{O})/I_{101}(\text{LiNiO}_2)$ 0.003

10 $I_{002}(\text{Li}_2\text{CO}_3)/I_{101}(\text{LiNiO}_2)$ 0.009

Half-width 003 reflection 0.1

Half-width 004 reflection 0.13

15 Example 3

Example 2 is repeated, wherein the mixture is initially calcined for 2 hours at 640°C under nitrogen and then for 30 minutes at 640°C under air.

- 20 The following values were obtained from X-ray diffraction analysis:

I_{104}/I_{003} (LiNiO₂) 0.76

$I_{111}(\text{Li}_2\text{O})/I_{101}(\text{LiNiO}_2)$ 0.037

$I_{002}(\text{Li}_2\text{CO}_3)/I_{101}(\text{LiNiO}_2)$ 0.017

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Half-width 003 reflection 0.17

Half-width 004 reflection 0.19

Claims

1. A process for preparing lithium transition metallates of the general formula



wherein

- 10 M^1 represents nickel, cobalt or manganese,
- M^2 represents chromium, cobalt, iron, manganese, molybdenum or aluminium and is not identical to M^1 ,
- 15 n is 2 if M^1 is manganese, otherwise 1,
- x is a number between 0.9 and 1.2,
- y is a number between 0.5 and 1.0 and
- 20 z is a number between 1.9 and 2.1,

25 by calcining an intimate mixture of oxygen-containing transition metal compounds and an oxygen-containing lithium compound, which has been obtained by treating a solid powdered transition metal compound with a solution of the lithium compound and drying, characterised in that at least the M^1 compound is used in the form of a powder with a specific surface area of at least 20 m²/g (BET) and calcination is performed in a moving bed.

- 30 2. A process according to Claim 1, characterised in that the transition metallate is milled and sieved after calcination and the finer fraction from sieving is recycled to the moving bed.
- 35 3. A process according to Claim 1 or 2, characterised in that a mixed transition metal compound which contains at least some of the M^2 compound is used as the M^1 compound.

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4. A process according to one of Claims 1 to 3, characterised in that the solution of lithium compound contains at least some of the M^2 compound.
5. A process according to one of Claims 1 to 4, characterised in that calcination is performed in a rotary kiln, in a fluidised bed or in a fall-shaft reactor (downer).
6. A process according to one of Claims 1 to 5, characterised in that following calcination, milling is performed and, after milling, further calcination is performed in an oxygen-containing atmosphere.
7. A process according to one of Claims 1 to 6, characterised in that $LiNO_3$ is used as the lithium compound and $Ni(OH)_2$ is used as the M^1 transition metal compound.
8. A process according to Claim 7, characterised in that the NO_2 released during calcination is recovered as nitric acid and is reacted with $LiOH$ to give $LiNO_3$ which is used as the lithium compound.
9. A process according to one of Claims 1 to 8, characterised in that the transition metal compound treated with the solution of a lithium compound is dried by spray drying or mixer granulation.

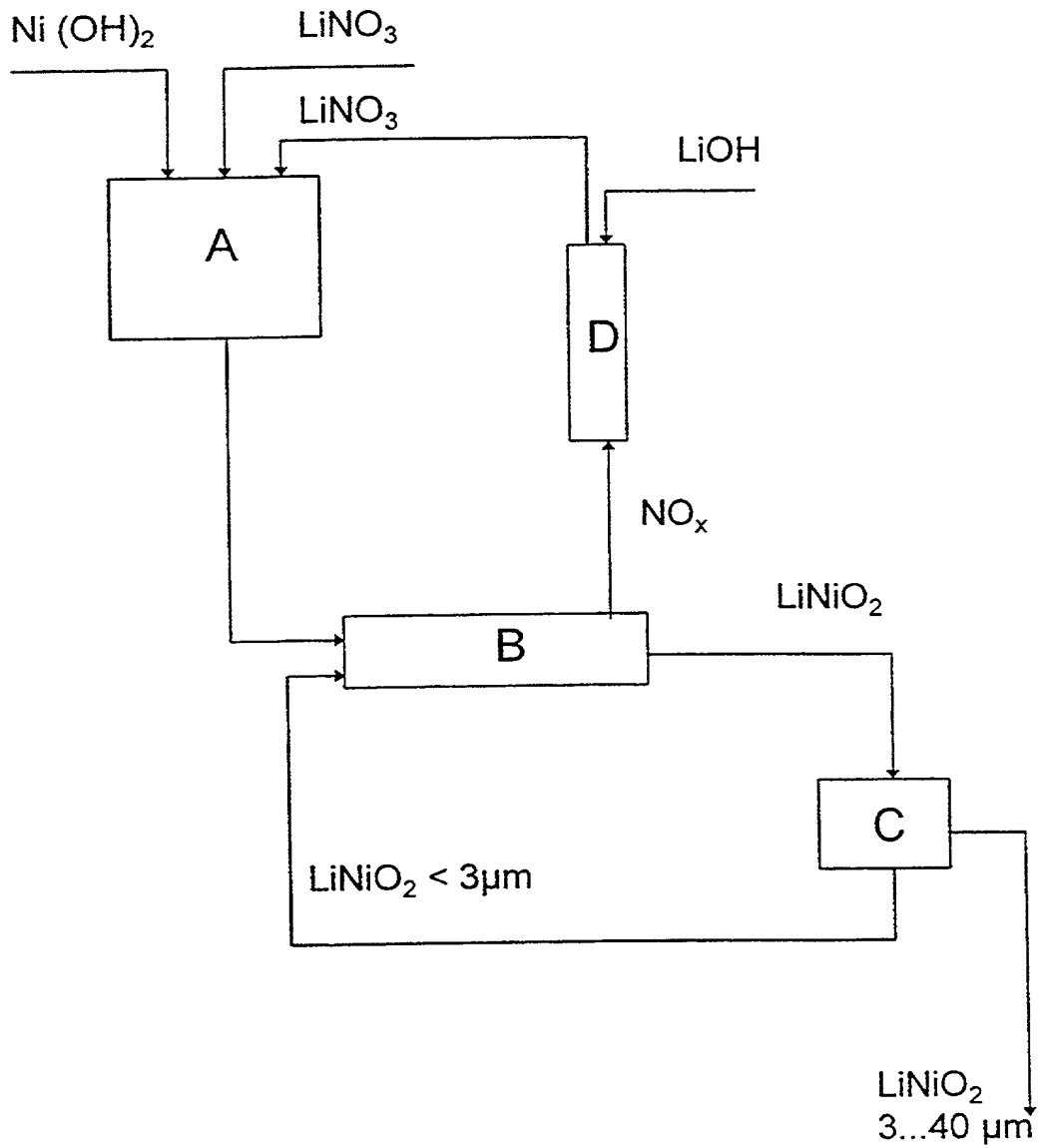


Fig. 1

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

"METHOD FOR PRODUCING LITHIUM-TRANSITION METAL MIXTURES"

the specification of which is attached hereto,

or was filed on **August 13, 1998**

as a PCT Application Serial No. **PCT/EP98/05150**

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s), the priority(ies) of which is/are to be claimed:

PCT/EP 98/00697
(Number)

EP
(Country)

February 9, 1998
(Month/Day/Year Filed)

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose the material information as defined in Title 37, Code of Federal Regulations, §1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)	(Filing Date)	(Status)
		(patented, pending, abandoned)

(Application Serial No.)	(Filing Date)	(Status)
		(patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

Jerry Cohen, Reg. No. 20,552
Edwin H. Paul, Reg. No. 31,405
Harvey Kaye, Reg. No. 18,978
Timothy J. Shea II, Reg. No. 37,504
Jacob N. Erlich, Reg. No. 24,338

Send Correspondence To:
PERKINS, SMITH & COHEN

One Beacon Street
Boston, Massachusetts 02108

Direct Telephone Calls To:

Jerry Cohen
(617) 854-4000

1-00	FULL NAME OF SOLE OR FIRST INVENTOR Mathias Benz	INVENTOR'S SIGNATURE <i>Mathias Benz</i>	DATE 28.6.00
	RESIDENCE D 38640 Goslar, Germany DEX	CITIZENSHIP German	
	POST OFFICE ADDRESS c/o H.C. Starck GmbH & Co. KG, Im Schleeke 78-91, D 38642 Goslar, Germany		
2-00	FULL NAME OF SECOND INVENTOR Wolfgang Kummer	INVENTOR'S SIGNATURE <i>Wolfgang Kummer</i>	DATE 29.6.00
	RESIDENCE D 38642 Goslar, Germany DEX	CITIZENSHIP German	
	POST OFFICE ADDRESS c/o H.C. Starck GmbH & Co. KG, Im Schleeke 78-91, D 38642 Goslar, Germany		
3-00	FULL NAME OF THIRD INVENTOR Evelyn Proß	INVENTOR'S SIGNATURE <i>Evelyn Proß</i>	DATE 24.07.00
	RESIDENCE D 79774 Albbruck, Germany DEX	CITIZENSHIP German	
	POST OFFICE ADDRESS c/o H.C. Starck GmbH & Co. KG, Im Schleeke 78-91, D 38642 Goslar, Germany		
4-00	FULL NAME OF FOURTH INVENTOR Josef Schmoll	INVENTOR'S SIGNATURE <i>Josef Schmoll</i>	DATE 29.06.00
	RESIDENCE D 38642 Goslar, Germany DEX	CITIZENSHIP German	
	POST OFFICE ADDRESS c/o H.C. Starck GmbH & Co. KG, Im Schleeke 78-91, D 38642 Goslar, Germany		
5-00	FULL NAME OF FIFTH INVENTOR Wolfgang Schweda	INVENTOR'S SIGNATURE <i>Wolfgang Schweda</i>	DATE 30.6.00
	RESIDENCE D 38723 Rhüden, Germany DEX	CITIZENSHIP German	
	POST OFFICE ADDRESS c/o H.C. Starck GmbH & Co. KG, Im Schleeke 78-91, D 38642 Goslar, Germany		
6-00	FULL NAME OF SIXTH INVENTOR Daniel Duff	INVENTOR'S SIGNATURE <i>D. G. Duff</i>	DATE 26.6.00
	RESIDENCE D 51373 Leverkusen, Germany	CITIZENSHIP German-British D9D	
	POST OFFICE ADDRESS c/o BAYER AKTIENGESELLSCHAFT, D 51368 Leverkusen, Germany		
7-00	FULL NAME OF SEVENTH INVENTOR Ricarda Leiberich	INVENTOR'S SIGNATURE <i>Ricarda Leiberich</i>	DATE 26.6.00
	RESIDENCE D 63225 Langen, Germany DEX	CITIZENSHIP German	
	POST OFFICE ADDRESS c/o BAYER AKTIENGESELLSCHAFT, D 51368 Leverkusen, Germany		
8-00	FULL NAME OF EIGHTH INVENTOR Christoph Schild	INVENTOR'S SIGNATURE <i>Christoph Schild</i>	DATE 26.06.2000
	RESIDENCE D 51373 Leverkusen, Germany DEX	CITIZENSHIP German	
	POST OFFICE ADDRESS c/o BAYER AKTIENGESELLSCHAFT, D 51368 Leverkusen, Germany		

9-00	FULL NAME OF NINTH INVENTOR Ulrich Krynitz	INVENTOR'S SIGNATURE <i>Ulrich Krynitz</i>	DATE 31.05.00
	RESIDENCE D 38640 Goslar, Germany DEX	CITIZENSHIP German	
	POST OFFICE ADDRESS c/o H.C. Starck GmbH & Co. KG, Im Schleeke 78-91, D 38642 Goslar, Germany		
10-00	FULL NAME OF TENTH INVENTOR Juliane Meese-Marktscheffel	INVENTOR'S SIGNATURE <i>Juliane Meese-Marktscheffel</i>	DATE 4.7.2000
	RESIDENCE D 38640 Goslar, Germany DEX	CITIZENSHIP German	
	POST OFFICE ADDRESS c/o H.C. Starck GmbH & Co. KG, Im Schleeke 78-91, D 38642 Goslar, Germany		
11-00	FULL NAME OF ELEVENTH INVENTOR Viktor Stoller	INVENTOR'S SIGNATURE <i>Viktor Stoller</i>	DATE 30.6.2000
	RESIDENCE D 38667 Bad Harzburg, Germany DEX	CITIZENSHIP German	
	POST OFFICE ADDRESS c/o H.C. Starck GmbH & Co. KG, Im Schleeke 78-91, D 38642 Goslar, Germany		
	FULL NAME OF TWELFTH INVENTOR	INVENTOR'S SIGNATURE	DATE
	RESIDENCE	CITIZENSHIP	
	POST OFFICE ADDRESS		
	FULL NAME OF THIRTEENTH INVENTOR	INVENTOR'S SIGNATURE	DATE
	RESIDENCE	CITIZENSHIP	
	POST OFFICE ADDRESS		
	FULL NAME OF FOURTEENTH INVENTOR	INVENTOR'S SIGNATURE	DATE
	RESIDENCE	CITIZENSHIP	
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	FULL NAME OF FIFTEENTH INVENTOR	INVENTOR'S SIGNATURE	DATE
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	RESIDENCE	CITIZENSHIP	
	POST OFFICE ADDRESS		